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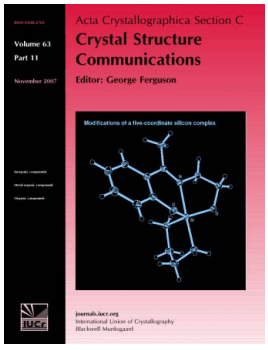

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Bis[(*S*)-(−)-α-methylbenzyl]thiourea

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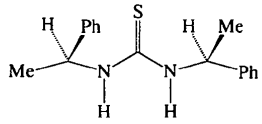
Abstract

The title compound, 1,3-bis[(*S*)-(−)-1-phenylethyl]thiourea, $C_{17}H_{20}N_2S$, exhibits bond lengths and angles consistent with the build-up of negative charge on the S atom. There are weak hydrogen bonds between molecules leading to helices disposed about crystallographic 3_1 axes.

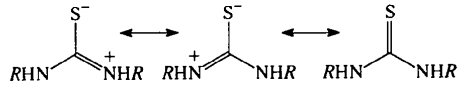
Comment

Our recent interest in the application of guanidines as ligands for transition metals has lead us to isolate a number of different thiourea compounds as intermediates in their synthesis (Bailey, Blake, Kryszczuk, Parsons & Reed, 1995). We report here the crystal structure of one of these, namely, enantiomerically pure bis[(*S*)-(−)-α-methylbenzyl]thiourea, (1), which was synthesized *via* standard procedures from (*S*)-(−)-α-methylbenzylamine and carbon disulfide (Yamazaki, Higashi & Iguchi, 1974).



(1)

The crystal structure is composed of discrete molecules of (1) (Fig. 1), the bond lengths and angles (Table 1) being quite typical for acyclic thiourea compounds in the Cambridge Structural Database (Allen & Kennard, 1993; Fletcher, McMeeking & Parkin, 1996). The C—S bond length [1.710 (7) Å] lies at the low end of the range associated with C—S single bonds (1.70–1.80 Å; Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1992), while the average C—N bond length [1.321 (8) Å] is similar to 'standard' C—N bonds in aromatic rings [1.336 (14) Å; Allen *et al.*, 1992]. Compound (1) should thus be properly regarded as a resonance hybrid of C=N-containing canonical forms, with only a relatively small contribution from the C=S-bonded form frequently used to represent thiourea compounds (see scheme below). The phenyl groups adopt a pseudo-antiperiplanar arrangement in order to minimize steric crowding of the α-methylbenzyl functionality.



R = –CH(Me)Ph

Hydrogen bonds link molecules to form a helix around the crystallographic threefold screw axis. Each molecule is linked to its neighbour in the helix by two N—H···S interactions [N1*A*···S^{i} 3.411 (6) and N1*B*···S^{i} 3.545 (7) Å; symmetry code: (i) $2-y$, $1+x-y$, $\frac{1}{3}+z$]. There appear to be no significant contacts between the helices.

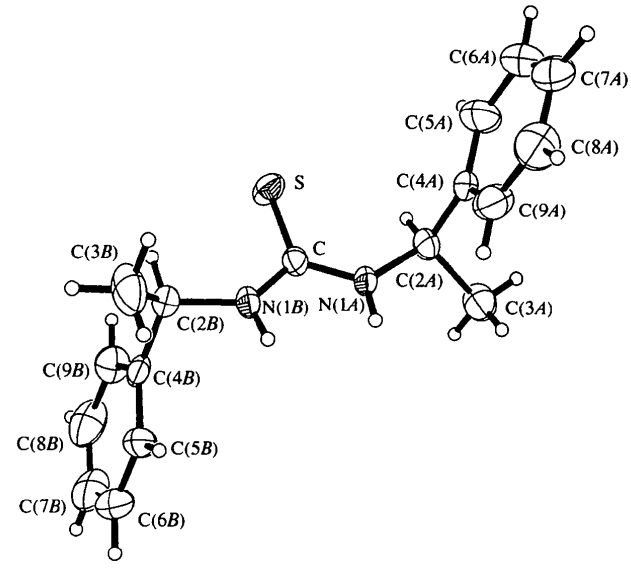


Fig. 1. A view of the title molecule with the atom-numbering scheme. Displacement ellipsoids enclose 30% probability surfaces. The dihedral angle between the planes of the phenyl rings is 11.6 (4)°.

Experimental

Crystals of (1) were grown from dichloromethane layered with diethyl ether. At 1.08 mm, the crystal selected for study is long by normal standards, but the crystals shattered when attempts were made to cut them. A 1.2 mm diameter collimator was used during data collection.

Crystal data

$C_{17}H_{20}N_2S$	Mo $K\alpha$ radiation
M_r = 284.41	λ = 0.71073 Å
Trigonal	Cell parameters from 56 reflections
$P3_1$	θ = 12.5–14.0°
a = 10.3144 (12) Å	μ = 0.193 mm^{-1}
c = 13.177 (2) Å	T = 296 (2) K
V = 1214.1 (3) Å^3	Needle
Z = 3	1.08 × 0.16 × 0.16 mm
D_x = 1.167 Mg m^{-3}	Colourless
D_m not measured	

Data collection

Stoe Stadi-4 four-circle diffractometer	R_{int} = 0.0451
ω–θ scans with width (1.32 + 0.35tanθ)°	θ_{max} = 22.51°
Absorption correction: none	h = −9 → 9
1890 measured reflections	k = 0 → 11
1053 independent reflections	l = 0 → 14
719 reflections with $I > 2\sigma(I)$	3 standard reflections frequency: 60 min intensity decay: none

Refinement

Refinement on F^2	Extinction correction: *SHELXTL* (Sheldrick, 1995)
$R[F > 4\sigma(F)]$ = 0.0456	Extinction coefficient: 0.008 (2)
$wR(F^2)$ = 0.0788	Scattering factors from *International Tables for Crystallography* (Vol. C)
S = 1.018	Absolute configuration: Flack (1983)
1053 reflections	Flack parameter = 0.18 (17)
192 parameters	
H atoms: see below	
$w = 1/[\sigma^2(F_o^2) + (0.0284P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$	
$(\Delta/\sigma)_{max}$ = −0.044	
$\Delta\rho_{max}$ = 0.136 e Å^{-3}	
$\Delta\rho_{min}$ = −0.154 e Å^{-3}	

Table 1. *Selected geometric parameters (Å, °)*

S—C	1.710 (7)	N1*A*—C2*A*	1.444 (8)
C—N1*A*	1.318 (8)	N1*B*—C2*B*	1.446 (8)
C—N1*B*	1.324 (7)		
N1*A*—C—N1*B*	115.5 (6)	C—N1*A*—C2*A*	127.3 (6)
N1*A*—C—S	121.4 (5)	C—N1*B*—C2*B*	128.7 (7)
N1*B*—C—S	123.1 (6)		

The crystal diffracted only weakly at high angle, so data were not collected beyond θ = 22.5°. H atoms attached to C atoms were placed in calculated positions and subsequently allowed to ride on their parent atoms with U_{iso}(H) = 1.2U_{eq}(C). Methyl groups were treated as rotating rigid groups with U_{iso}(H) = 1.5U_{eq}(C). H atoms attached to N atoms were located in a difference synthesis; the positional parameters were refined subject to similarity restraints on the N—H distances and the isotropic displacement parameters refined freely.

Data collection: *DIF*4 (Stoe & Cie, 1990*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1990*b*). Program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994). Program(s) used to refine structure: *SHELXTL* (Sheldrick, 1995). Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL*.

The authors thank the EPSRC for provision of a four-circle diffractometer and for use of the Chemical Database at Daresbury.

Lists of structure factors, anisotropic displacement parameters, atomic coordinates and complete geometry have been deposited with the IUCr (Reference: HA1175). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2′,3′-Diphenyl-Substituted *p*-Terphenyls

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Abstract

The crystal structure analyses of the title substituted *p*-terphenyls, 2′-(*p*-methoxyphenyl)-3′-phenyl-*p*-terphenyl, $C_{31}H_{24}O$, and 2′,3′-bis(*p*-methoxyphenyl)-*p*-